Update on ESTCP Project ER-0918: Field Sampling and Sample Processing for Metals on DoD Ranges

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Project Team

- Jay L. Clausen, PI: ERDC-CRREL
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- Thomas Georgian: HNC@EMCX
- Larry Penfold: Test America
- Diane Anderson: APPL Laboratories

Technical Objectives

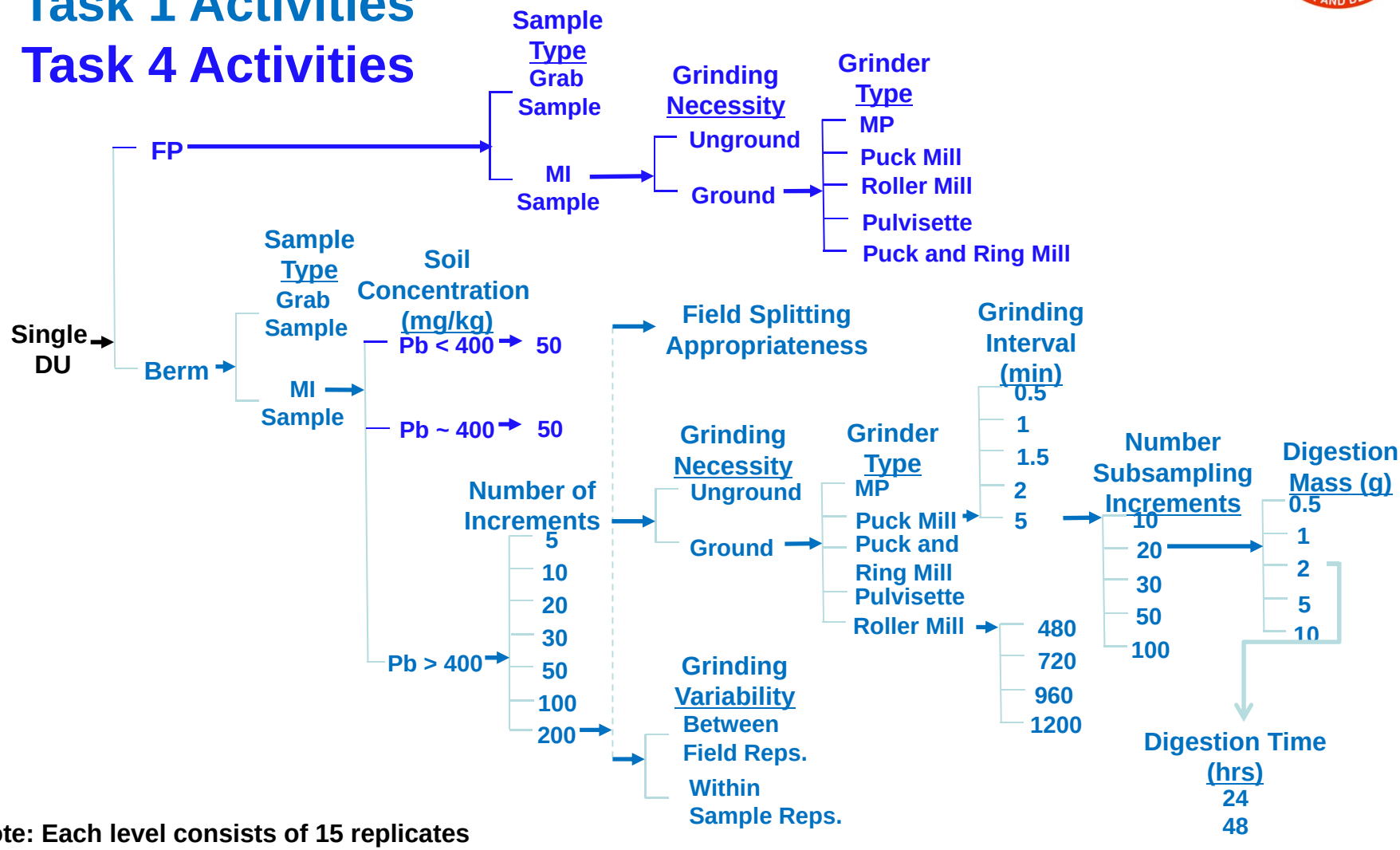
- Demonstrate improved data quality for metal constituents in surface soils on military training ranges by coupling multi-increment sampling with modifications to sample preparation and analysis methods such as:
  - Sample processing involving grinding
  - Sub-sampling to build the digestate aliquot
  - Digestion Issues (mass, acid ratio, time)
  - Laboratory processing protocol applicable to both metals and energetics

Experimental Design –Task 1

- Multi-increment versus grab samples
- Number of increments per decision unit
- Grinding necessity
- Digestion mass evaluation
- Digestion time
- Blank material identification and assessment
- Puck Mill metal carry over assessment (cross contamination)
- Grinder comparisons
- Puck Mill and Roller Mill optimum grinding interval
- Appropriateness of field splitting
- Subsampling for digestate preparation

Experimental Design –Task 1

Task 1 Activities
Task 4 Activities



Note: Each level consists of 15 replicates

Soil Test Material

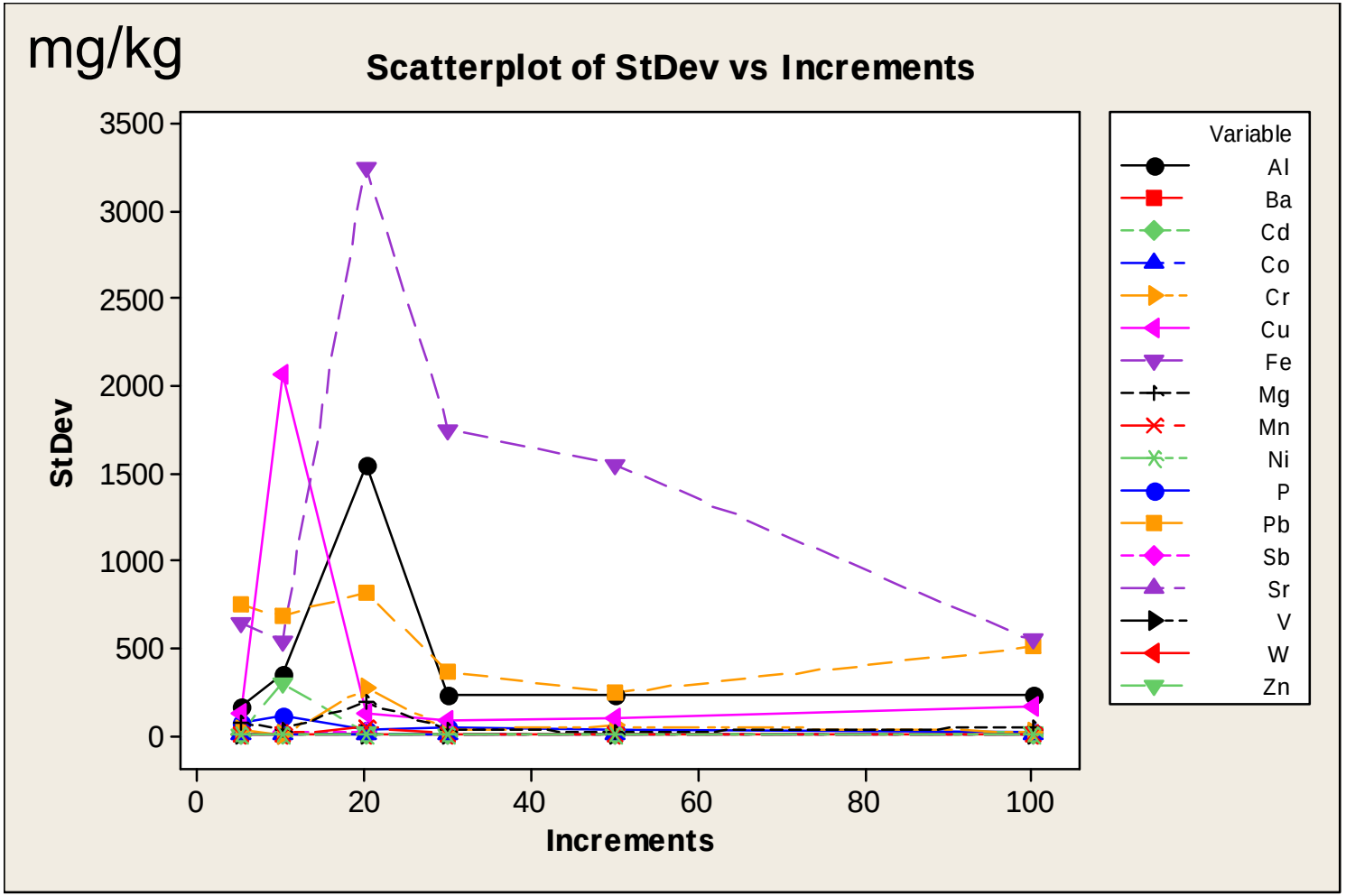
- Site: Camp Ethan Allen, VT
- Range Type: Small Arms (Pistol, Rifle)
- Decision Unit: Berm Face – 3 by 30 m
- Soil Type: Silty sand, low CEC, low OM, pH~ 5
- Metal Content: 100's to low 1,000's ppm
- Samples Collected
  - Grab/discrete using grid-node approach – 30
  - Multi-increment using systematic random, 7 replicates of 5, 10, 20, 30, 50, 100 increments
  - One 200 increment sample ~ 25 kg

Soil Test Material

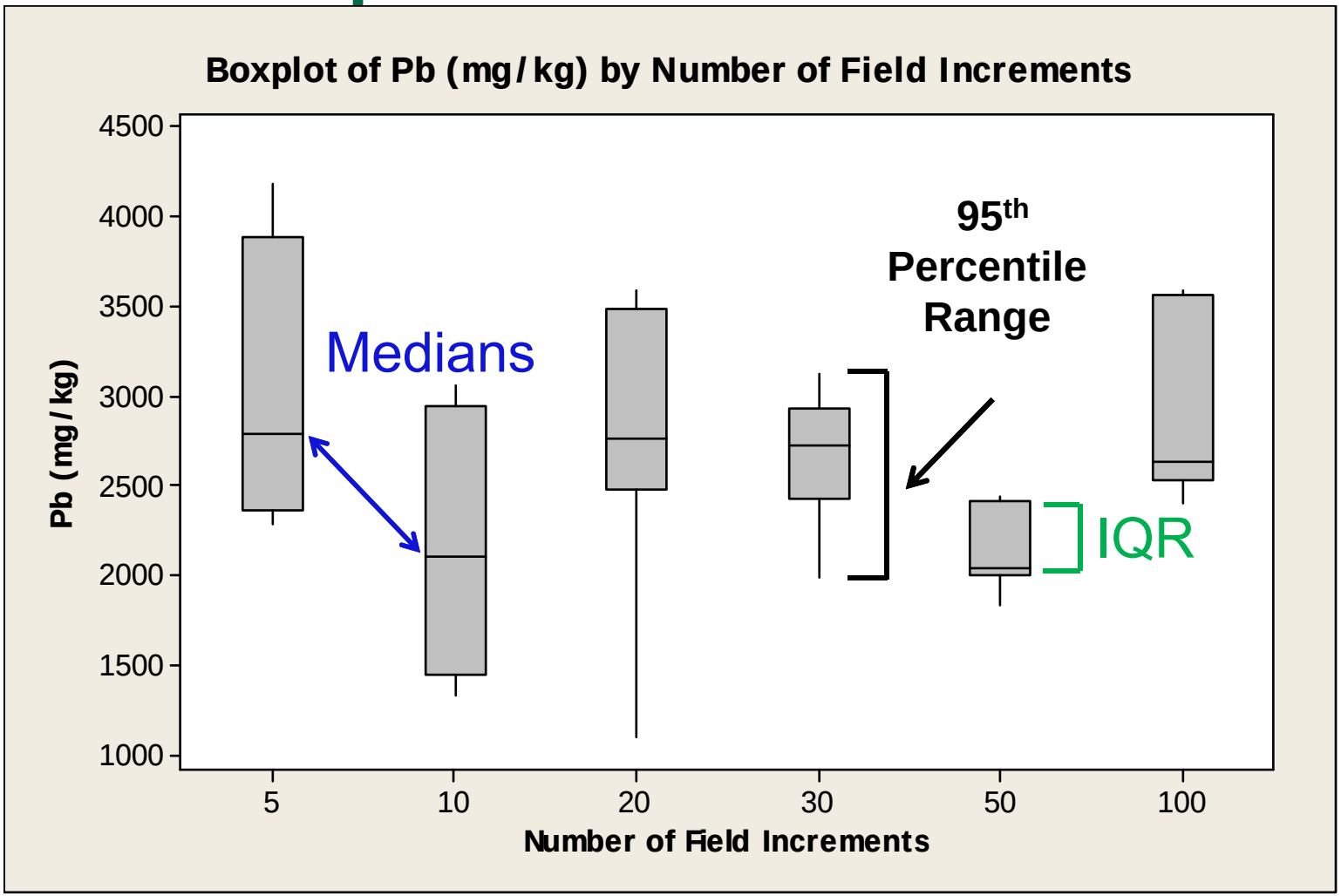
Multi-Increment vs Grab Samples

		Sb mg/kg	Cu mg/kg	Pb mg/kg	Zn mg/kg
Grab	Mean	88	300	5060	66
(n=30)	Std. Dev.	375	132	14,437	17.5
	RSD (%)	**426**	**44**	**285**	**27**
MI-30	Mean	23	573	2664	67
(n=7)	Std. Dev.	3.3	85	367	4.0
	RSD (%)	**14**	**15**	**14**	**6**
MI-50	Mean	17.6	457	2156	67
(n=7)	Std. Dev.	1.8	96	243	6.5
	RSD (%)	**10**	**21**	**11**	**10**

Number of Increments per Decision Unit



Number of Increments per Decision Unit



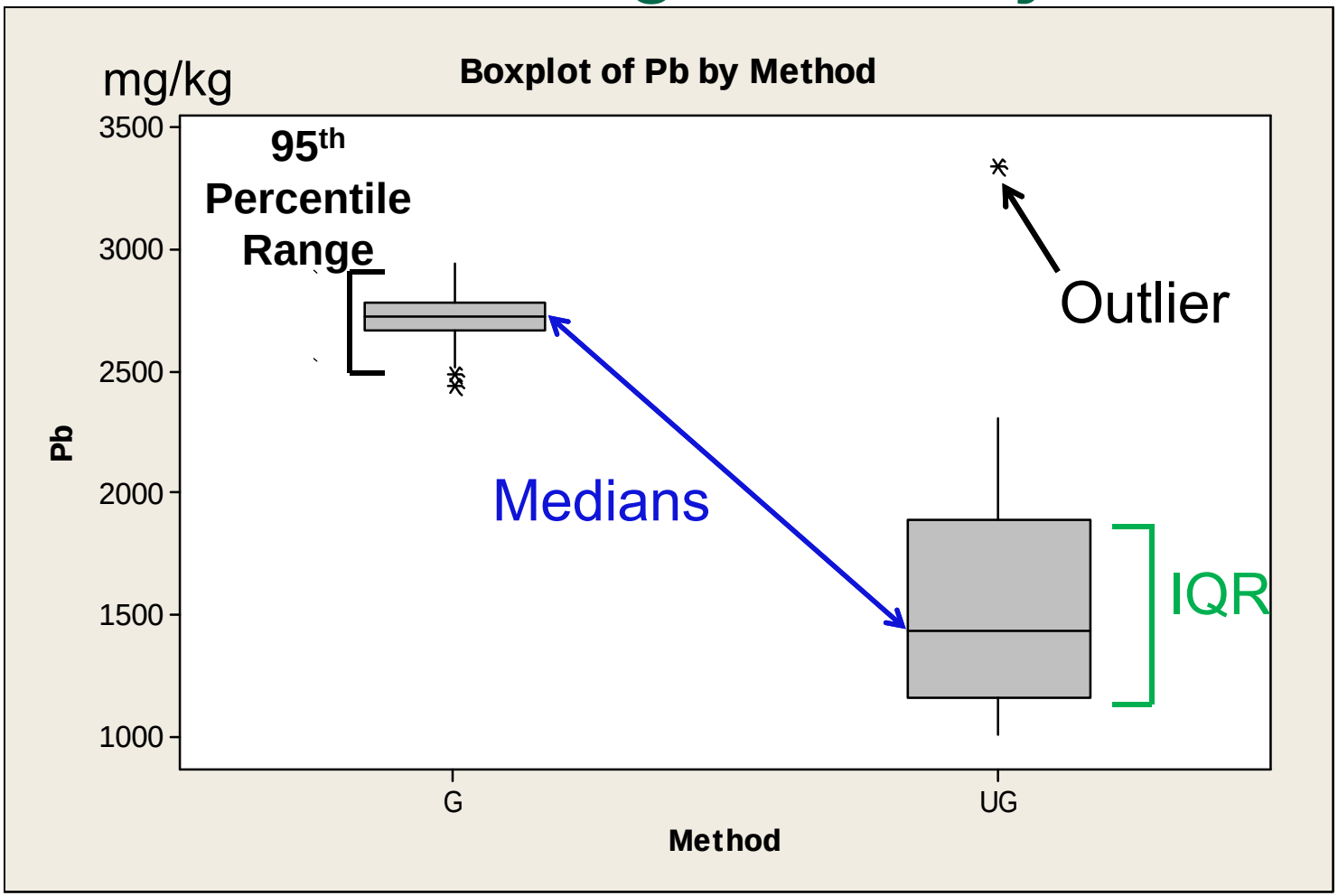
Grinding Necessity

		Sb mg/kg	Cu mg/kg	Pb mg/kg	Zn mg/kg
Un-Ground	Mean	**14**	**360**	**1600**	**66**
(n=15)	Std. Dev.	**10**	**90**	**630**	**11.3**
	RSD (%)	**71**	**25**	**39**	**17**
Ground	Mean	**23**	**550**	**2720**	**77**
(n=15)	Std. Dev.	**1.6**	**100**	**120**	**8.7**
	RSD (%)	**7.0**	**18**	**4.4**	**11**

Performance criteria RSD < 15% for lab replicates (for concentrations > 100

Soil Post Grinding

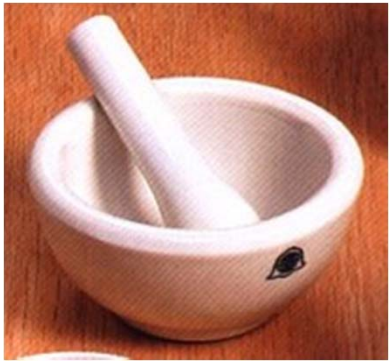
Grinding Necessity



Performance Assessment – Sample Processing (Grinding) of Soil

Puck Mill

Fe, Mn, Cr, V



Mortar and Pestle
Ceramic

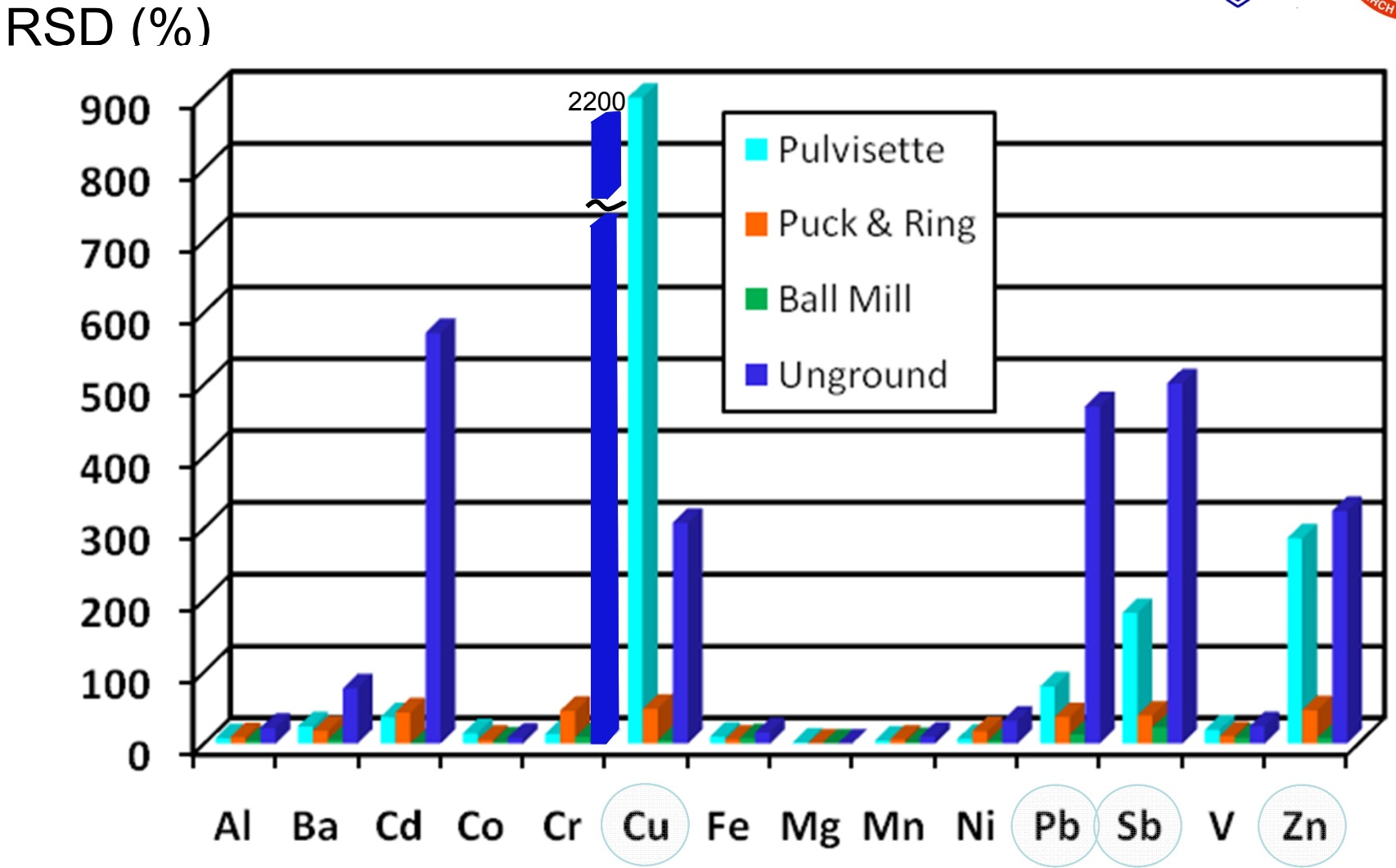
Roller Mill

Alumina cans polyethylene
Liner, ceramic balls

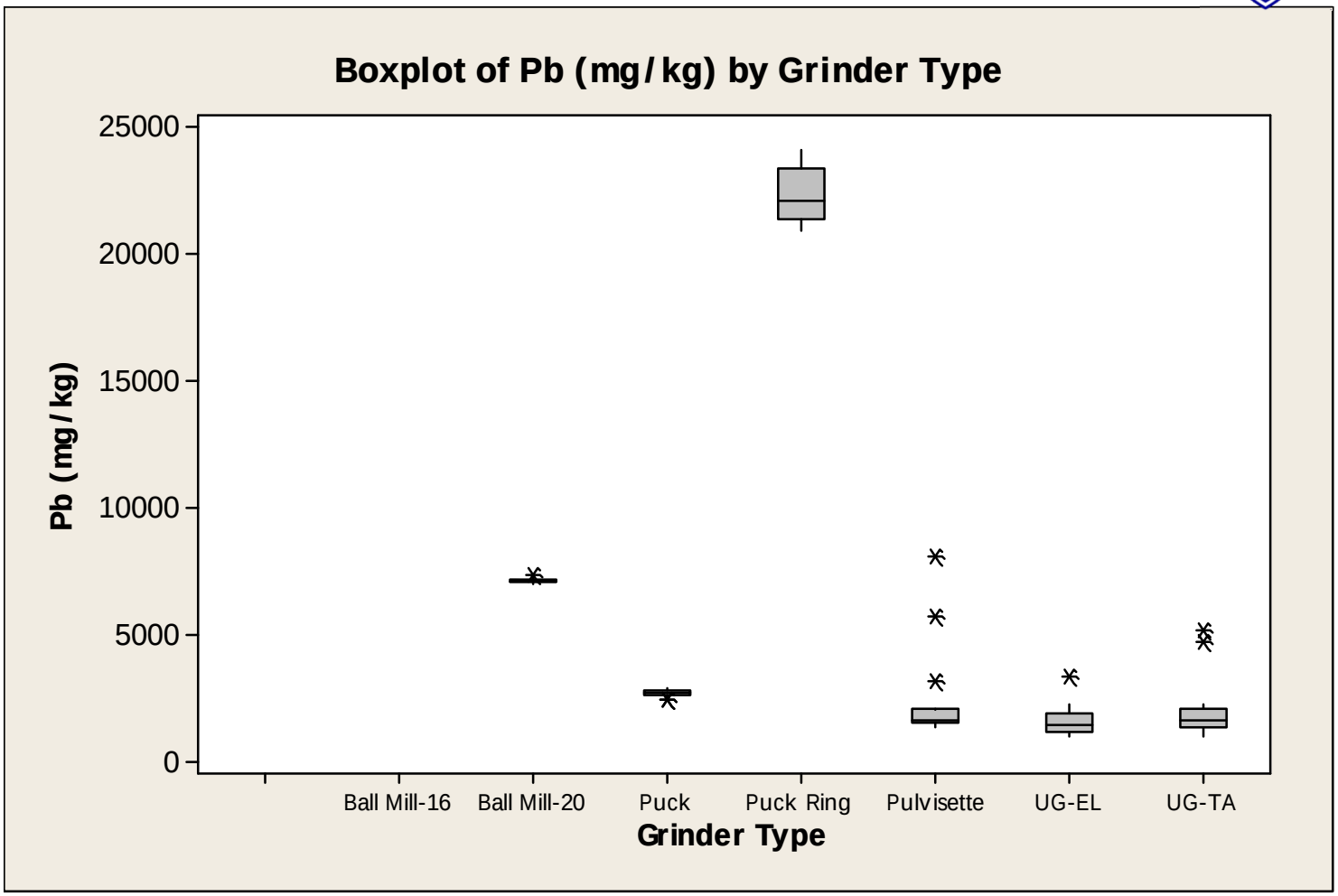
Pulvisette

Agate balls

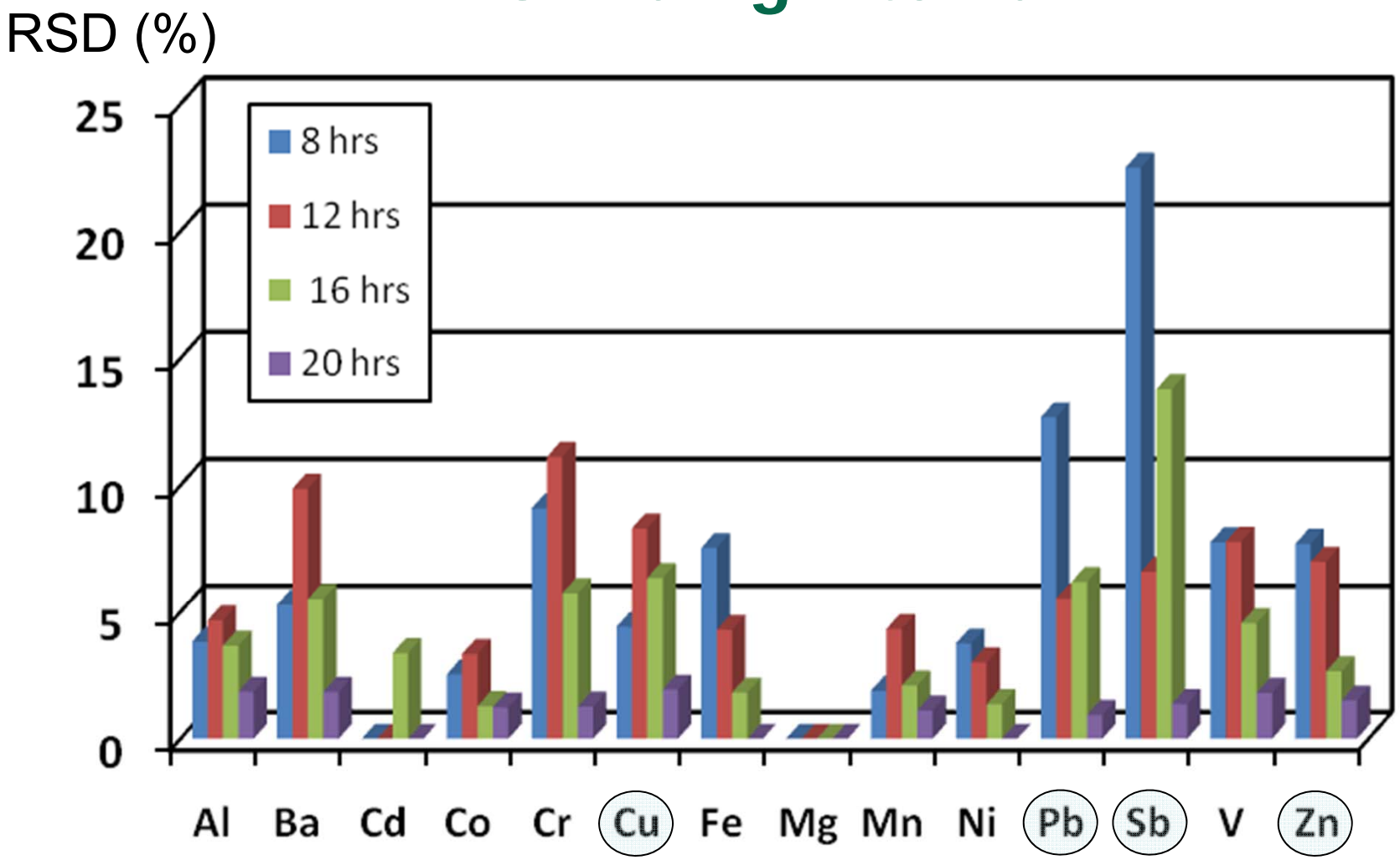
Grinder Comparisons



Grinder Comparisons



Roller Mill Optimum Grinding Interval



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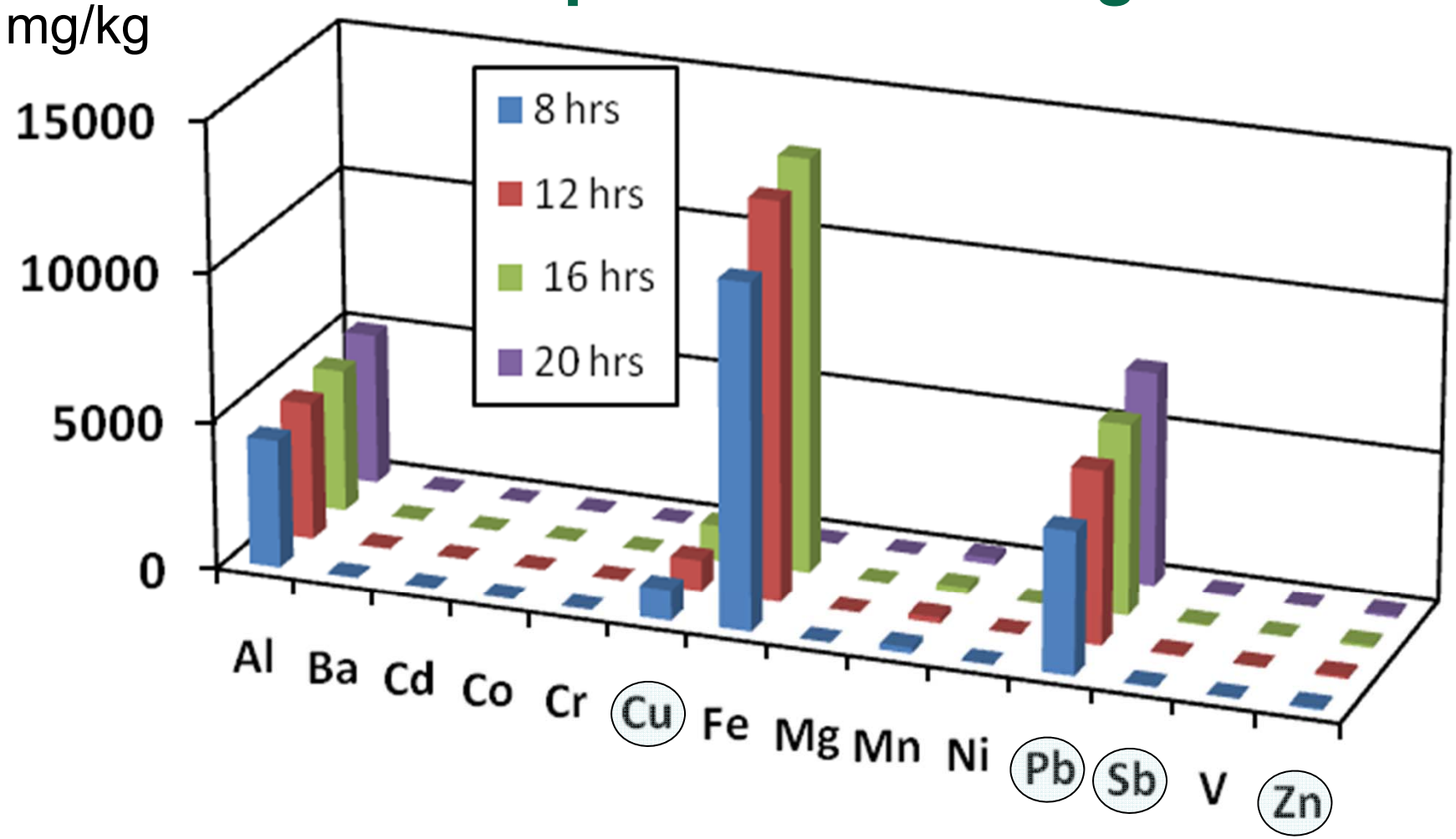

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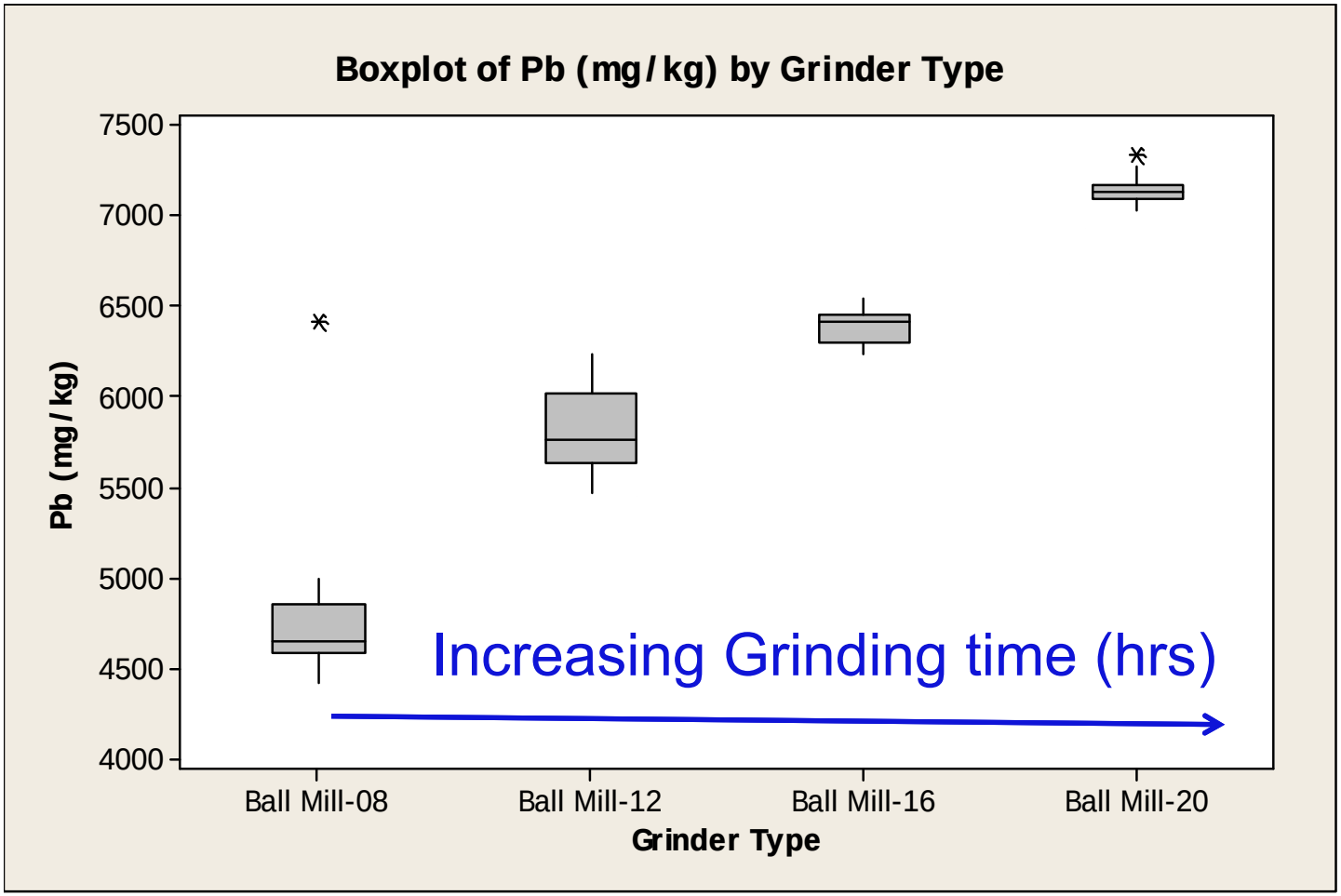
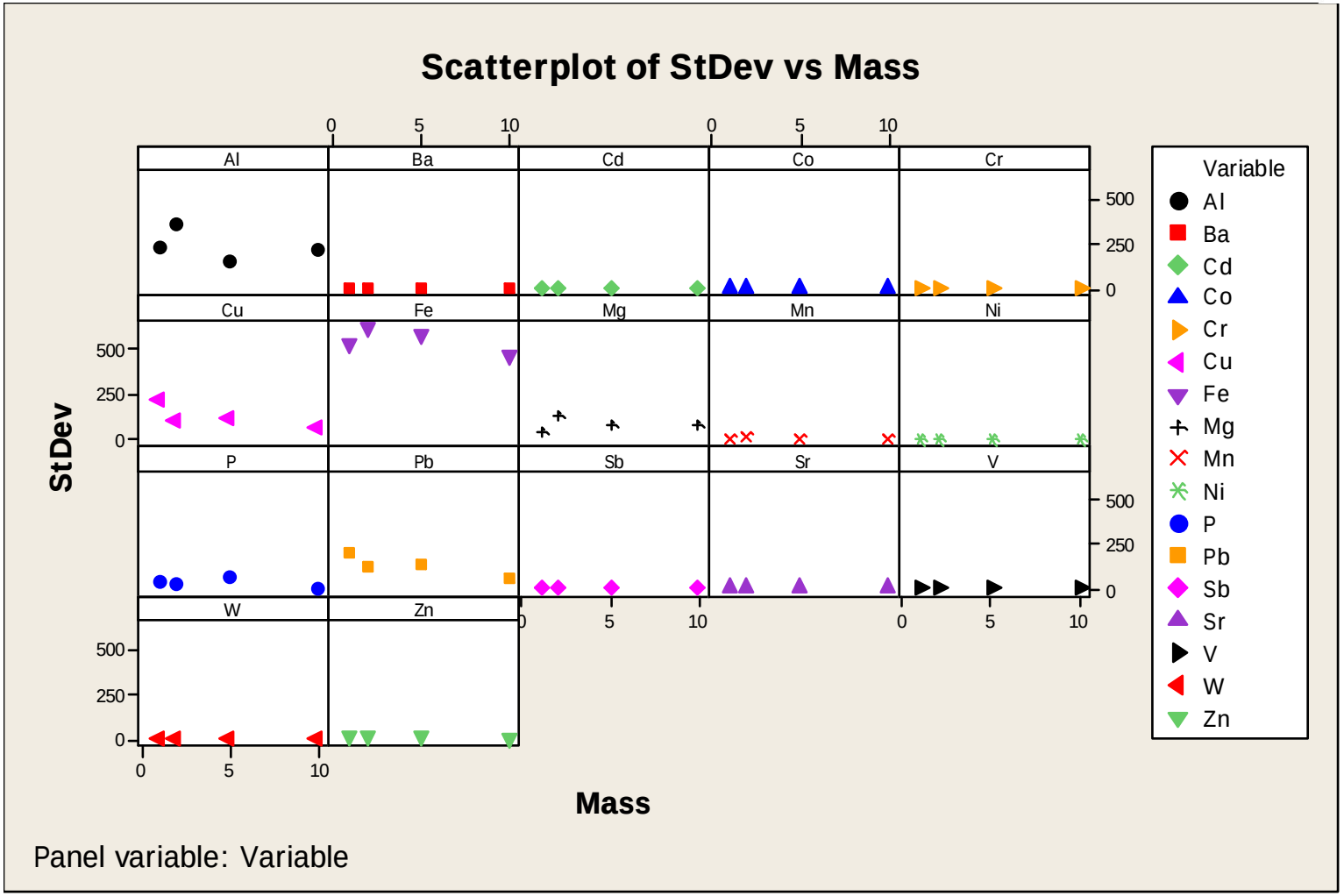


Digestion Mass



Digestion Time

Metal	M_{24} (mg/kg)	M_{48} (mg/kg)	Metal	M_{24} (mg/kg)	M_{48} (mg/kg)
Al	5678	6075	Mn	223.9	**242.8**
Ba	30.29	**32.09**	Ni	**12.24**	11.67
Cd	**1.825**	1.050	P	612.3	**630.0**
Co	8.60	**8.935**	Pb	2718	**2893**
Cr	221.2	**242.1**	Sb	**22.61**	20.59
Cu	542.5	498.2	Sr	21.51	**23.80**
Fe	16920	**17293**	V	15.14	**16.32**
Mg	2121	**2259**	Zn	75.80	79.88

M_{24} , M_{48} = Median 24- and 48-hr digestions, respectively

Issues

- Analysis error is still greater than expected between laboratories, believed associated with volume of acid used during digestion
- Considerable mass of metal remains in over size fraction (typically discarded)

- Ongoing question of impact of sample preparation method changes to risk determination
- Poor recovery of antimony is evident with conventional analysis; new digestion process needed